

# UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.

199764US3

First Inventor or Application Identifier

Takayuki TOSHIMA, et al.

Title

SILYLATION TREATMENT UNIT AND METHOD

## APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents

Fee Transmittal Form (e.g. PTO/SB/17)

(Submit an original and a duplicate for fee processing)

Specification

Total Sheets **37**

Drawing(s) (35 U.S.C. 113)

Total Sheets **14**  
(Formals)

4. ☒ Oath or Declaration

Total Pages **4**

a. ☒ Newly executed (original)

b. ☐ Copy from a prior application (37 C.F.R. §1.63(d))  
(for continuation / divisional w/ box 16 completed)

i. ☐ DELETION OF INVENTOR(S)

Signed statement attached deleting inventor(s) named in  
the prior application, see 37 C.F.R. §1.63(d)(2) and  
1.53(d).

5. ☐ CD-ROM or CD-R in duplicate, large table or Computer  
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## ACCOMPANYING APPLICATION PARTS

7. ☒ Assignment Papers (cover sheet & document(s))

8. ☐ Application Data Sheet. See 37 CFR 1.76

9. ☐ 37 C.F.R. §3.73(b) Statement ☐ Power of Attorney  
(when there is an assignee)

10. ☐ English Translation Document (if applicable)

11. ☐ Information Disclosure  
Statement (IDS)/PTO-1449 ☐ Copies of IDS  
Citations

12. ☐ Preliminary Amendment

13. ☒ White Advance Serial No. Postcard

14. ☐ Certified Copy of Priority Document(s)  
(if foreign priority is claimed)

15. ☐ Applicant claims small entity status.  
See 37 CFR 1.27

16. ☒ Other: Notice of Priority

16. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application no.:

Prior application information: Examiner:

Group Art Unit:

For CONTINUATION OR DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is  
considered a part of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon  
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17. Amend the specification by inserting before the first line the sentence:

☐ This application is a ☐ Continuation ☐ Division ☐ Continuation-in-part (CIP)  
of application Serial No. Filed on

☐ This application claims priority of provisional application Serial No.

Filed

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Docket No. 199764US3

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

INVENTOR(S) Takayuki TOSHIMA, et al.

SERIAL NO: New Application

FILING DATE: Herewith

FOR: SILYLATION TREATMENT UNIT AND METHOD

**FEE TRANSMITTAL**

ASSISTANT COMMISSIONER FOR PATENTS  
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Respectfully Submitted,

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# TITLE OF THE INVENTION

## SILYLATION TREATMENT UNIT AND METHOD

### CROSS-REFERENCE TO RELATED APPLICATIONS

5 This application is based upon and claims the benefit of priority from the prior Japanese Patent Application No. 11-328269, November 18, 1999, the entire contents of which are incorporated herein by reference.

### BACKGROUND OF THE INVENTION

10 The present invention relates to a silylation treatment unit and a silylation treatment method for performing a silylation treatment on a surface of a substrate, such as a semiconductor wafer and an LCD substrate.

15 In manufacturing a microelectronic device such as a semiconductor integrated circuit, more severe performance is required for a lithography technology and a resist material which are used for the processing, as a pattern processed on a silicon wafer becomes finer.

20 Regarding to the lithography technology used for manufacturing a device, a wavelength of a light source which is used for an exposure of the pattern is becoming shorter and an i-ray and a KrF excimer laser light come to be used.

25 The lithography is performed with the i-ray by using a photosensitizer of a novolac resist as a base resin. However, when using an ArF excimer laser light

as the result of the shorter wavelength, the fineness can not be achieved because the novolac resist has a high light absorption characteristic. Therefore, a resist using a phenolic ring compound is proposed.

5 Although the phenolic resist like this has an advantage that a plasma resistance increases, the phenolic resist has an extremely high light absorptance, and its tendency is growing as the wavelength becomes shorter. Particularly, the light does not reach the enough depth  
10 when the ArF excimer laser light is used.

A silylation method is spotlighted as a method having the enough sensibility and improving the plasma resistance, even when the light source of the short wavelength, such as the ArF excimer laser light is used.

15 With this silylation method, a resist pattern having the enough selectivity can be formed by exposing the photosensitizer in a predetermined pattern image, performing a silylation on the surface of thus-exposed photosensitizer, and performing a dry developing using  
20 the silylation treated photosensitizer as a mask.

Based on the conventional silylation treatment method, there is a problem to be solved as explained below.

There is a problem that a silylation reaction  
25 actualizing the silylation method has an extremely high temperature dependency, in which the silylation reaction progresses ununiformly within the surface of a

wafer if the temperature within the surface of the wafer is ununiform. Therefore, it is necessary to obtain a uniformity of a silylation layer in order to employ the silylation method. To solve this problem, various measures have been taken conventionally by devising the hardware structures, such as the structure of a treatment chamber, a supplying method of a silylation atmosphere, and a precision of a hot plate. However, even though the uniformity of the silylation layer can be obtained by these measures, a minute defect in the hardware structure prevents the uniform formation of the silylation layer, since its processing condition depends on the hardware.

#### BRIEF SUMMARY OF THE INVENTION

The present invention is made to solve the aforementioned problem and its object is to provide a silylation unit and a silylation treatment method which are capable of obtaining a uniform silylation layer without depending on the hardware structure.

According to a first aspect of the present invention, there is provided a silylation unit comprising a chamber, a heating mechanism provided in the chamber for heating a substrate, a supplying mechanism for supplying a vapor including a silylation reagent into the chamber, and a substrate holder for holding the substrate in the chamber, in which an interval between the heating mechanism and the

substrate is adjustable to at least three levels or more.

When structured as above, it is possible to receive the substrate in a condition where it is least influenced by a heat in the chamber by maximizing the interval from the heating mechanism, bring the interval comparatively closer to the heating mechanism to wait until the temperature inside the chamber obtains a high planer uniformity, and further bring it closer to the heating mechanism after a high planer uniformity is obtained such that the silylation reaction occurs. Thus, by holding the substrate at a predetermined interval to the heating mechanism until the heating by the heating mechanism becomes uniform, the silylation under the ununiform silylation atmosphere does not occur. Therefore, the uniform silylation layer can be obtained without depending on the hardware structure.

According to a second aspect of the present invention, there is provided a silylation treatment method comprising the steps of carrying in the substrate into the chamber for disposing at a predetermined interval from the heating mechanism provided in the chamber, supplying the vapor including the silylation reagent into the chamber such that the chamber is filled with the atmosphere of the silylation reagent, raising the temperature of the chamber by the heating mechanism, bringing the substrate closer to the

heating mechanism such that the silylation atmosphere is dispersed uniformly inside the chamber at the temperature where the silylation reaction of the substrate does not occur, and further bringing the substrate closer to the heating mechanism for making the temperature of the substrate higher such that the silylation reaction occurs on the surface of the substrate.

According to this method, it is preferable that the interval between the heating mechanism and the substrate is adjustable to at least three levels or more. Also, it is preferable that the substrate almost contacts the heating mechanism in the step of the silylation reaction.

Further, after the silylation treatment, the silylation treatment can be completed simply by supplying an inert gas into the chamber for replacing the vapor including the silylation reagent. Further, the excessive and ununiform silylation reaction can be prevented by spacing the interval between the heating mechanism and the substrate before replacing the gas inside the chamber.

Moreover, when being filled with the silylation atmosphere under the condition where the heating mechanism and the substrate are disposed at a predetermined interval, the volume of the gas decreases inside the chamber by reducing the pressure inside the

chamber so that the gas flow inside the chamber is  
stabled and the uniformity of the density of the  
silylation reagent further increases.

Furthermore, by making the silylation reaction to  
5 occur while stopping the supply of the silylation  
reagent into the chamber without exhausting from the  
chamber, the gas flow stops inside the chamber and the  
silylation reaction is made to occur while maintaining  
the uniform silylation atmosphere so that a planer  
10 uniformity of the silylation reaction on the wafer  
further increases.

Additional objects and advantages of the invention  
will be set forth in the description which follows, and  
in part will be obvious from the description, or may be  
15 learned by practice of the invention. The objects and  
advantages of the invention may be realized and  
obtained by means of the instrumentalities and  
combinations particularly pointed out hereinafter.

#### BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWING

20 The accompanying drawings, which are incorporated  
in and constitute a part of the specification,  
illustrate presently preferred embodiments of the  
invention, and together with the general description  
given above and the detailed description of the  
25 preferred embodiments given below, serve to explain the  
principles of the invention.

FIG. 1 is a top view showing the whole structure



of a silylation treatment unit according to a first embodiment of the present invention;

FIG. 2 is a vertical sectional view showing the whole structure of the silylation treatment unit according to the first embodiment;

FIG. 3 is a perspective view of a supply ring according to the first embodiment;

FIG. 4 is a view showing the whole structure of the silylation treatment unit according to the first embodiment, together with a control system;

FIG. 5 is a view of the whole structure of a resist treatment system having the silylation treatment unit according to the first embodiment;

FIG. 6 is a side view of the resist treatment system having the silylation treatment unit according to the first embodiment;

FIG. 7 is a front view for explaining the functions of the resist treatment system having the silylation treatment unit according to the first embodiment;

FIGS. 8A, 8B, and 8C are views showing a process of the silylation treatment according to the first embodiment;

FIG. 9 is a view showing a flow chart of the silylation treatment according to the first embodiment;

FIG. 10 is a view showing a flow chart of a modification of the silylation treatment;

FIG. 11 is a perspective view of another modification of the supply ring;

FIG. 12 is a plane view of still another modification of the supply ring;

5        FIG. 13 is an explanatory view of a side section showing the airflow when the supply ring in FIG. 12 is used;

FIG. 14 is an explanatory view of a side section explaining another modification of the silylation treatment unit according to the present invention;

10        FIG. 15 is an explanatory view of a side section explaining another modification of the silylation treatment unit according to the present invention; and

15        FIG. 16 is a perspective view of another modification of the supply ring.

#### DETAILED DESCRIPTION OF THE INVENTION

Hereinafter, preferred embodiments according to the present invention will be explained with reference to the attached drawings.

20        (First embodiment)

In this embodiment, a silylation treatment unit of the present invention which is applied to a resist treatment system for a semiconductor wafer will be explained.

25        FIGS. 1 and 2 are a plane view and a vertical sectional view showing the structure of the silylation treatment unit, respectively.

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As shown in FIG. 1, a silylation treatment unit 1 includes a base block 2. The base block 2 has a shape of hollow and comprises a base block side portion 2a which defines its side and a base block bottom portion 2b which defines its bottom. Further, a horizontal masking shield 3 is attached horizontally to the base block bottom portion 2b at a position of a predetermined height of the base block side portion 2a. A circular opening 4 is formed in the horizontal masking shield 3 and a hot plate 5 is received as a heating mechanism in this opening 4. The horizontal masking shield 3 supports the hot plate 5 by a supporting plate 6.

A treatment chamber 7 as a chamber for performing a silylation treatment is defined by the base block side portion 2a, the horizontal masking shield 3 and a cover 8. Openings 7A and 7B are respectively formed in the front side and the back side of the treatment chamber 7 and a wafer W is carried into/out of the treatment chamber 7 through the openings 7A and 7B.

Three holes 9 are penetratingly formed in the hot plate 5 and lifter pins 10 are respectively inserted through each of the holes 9 as wafer W holders. Three lifter pins 10 are connectively supported by an arm 11, and the arm 11 is connectively supported by, for example, a rod 12a of a vertical cylinder 12. When the rod 12a is projected from the vertical cylinder 12, the

lifter pins 10 are projected so as to lift the wafer W from the hot plate 5.

The height of the lifter pins 10 which support the wafer W at three points is adjustable to three levels such as low, medium and high (hereinafter each height is referred to as the low level, the medium level and the high level). At the low level, the lifter pins 10 do not project from the surface of the hot plate 5. Therefore, an interval between the wafer W which is held by the lifter pins 10 and the surface of the hot plate 5 is theoretically 0 mm, but the proximity in the actual structure of the unit is, for example, about 0.1 mm. At the medium level, the lifter pins 10 project from the surface of the hot plate 5, for example, by 7 mm. Further, at the high level, the lifter pins 10 project from the surface of the hot plate 5, for example, by 18 mm. The wafer W is transferred by a carrier mechanism which is not illustrated from another treatment mechanism at this high level.

As shown in FIG. 2, a ring-shaped shutter 13 is attached at the outer circumference of the hot plate 5. The shutter 13 is supported by a rod 16a of a vertical cylinder 16 through an arm 15 so that it is able to ascend and descend. This shutter 13 is retreated at a low level when the treatment is not performed, but rises when the treatment is performed to position

itself between the hot plate 5 and the cover 8. At the inner circumference of the shutter 13, a ring-shaped supply ring 14 is disposed in such a manner to surround the hot plate 5.

5           FIG. 3 shows the perspective structure of the supply ring 14 in detail. As shown in FIG. 3, the supply ring 14 includes an annular ring member 14b. Many supply holes 14a are formed along the inner circumference of the ring of the ring member 14b at  
10           pitch intervals of, for example, the central angle of  $2^\circ$ . Four supply paths 14c are opened at the bottom surface of the ring of this ring member 14b and at the symmetrical positions with respect to the center of the ring of the ring member 14b. These supply paths 14c  
15           communicate with a silylation reagent supply source (not illustrated) outside the base block 2 through the openings provided at the base block bottom portion 2b.

          In the center of the cover 8, an exhaust port 18 which communicates with an exhaust pipe 17 is opened.  
20           The gas generated in the heating treatment or the like is exhausted through this exhaust port 18. The exhaust pipe 17 communicates with a duct 19 (or 20) at the front side of the unit or another duct not illustrated.

          A machine room 21 is provided under the horizontal  
25           masking shield 3. The machine room 21 defines its periphery by the sidewall of the duct 19, a sidewall 22 and the base block bottom portion 2b. Inside the

machine room 21, for example, the hot plate supporting plate 6, the shutter arm 15, a lifter pin arm 11, an elevating cylinder 16, and the elevating cylinder 12 are provided.

5           As shown in FIG. 1, four protrusions 23, for example, are provided on the upper surface of the hot plate 5 and the wafer W is made to take its position by these four protrusions. Further, a plurality of small protrusions (not illustrated) are provided on the upper  
10       surface of the hot plate 5, where the tops of these small protrusions contact with the wafer W when the wafer W is mounted on the hot plate 5. Thus, a minute space (about 0.1 mm) is formed between the wafer W and the hot plate 5 so that the bottom surface of the wafer  
15       W is free from dust and scratch.

Next, a control system and a silylation reagent vapor supplying mechanism of the silylation treatment unit are explained with reference to FIG. 4.

As shown in FIG. 4, each of the supply paths 14c  
20       communicates with a silylation reagent vapor supply pipe 31 and this silylation reagent vapor supply pipe 31 supplies the silylation reagent vapor which is generated in a bubbler tank 32 into the treatment chamber 7. A mass flow controller 33 is provided to  
25       the silylation reagent vapor supply pipe 31, which controls the volume of the flow of the silylation reagent vapor supplied into the treatment chamber 7

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based on the control command from a controller 34.

5 A bubbling member 35 made of, for example, porous ceramic or the like is provided at the bottom surface of the bubbler tank 32, and a gas supply pipe 36 which  
10 supplies an inert gas such as  $N_2$  is inserted through the bubbling member 35. From the upper surface of the bubbler tank 32, a carrier gas, for example,  $N_2$  is supplied from a carrier gas supply pipe 37, the  
15 silylation reagent vapor is generated while supplying the inert gas from the gas supply pipe 36 into the bubbling member 35 to perform the bubbling of a silylation reagent 38 stored in the tank 32, and the silylation reagent vapor is supplied into the treatment chamber 7 from the gas supply pipe 31 by using  $N_2$  as the carrier gas.

20 The hot plate 5 has an electrical resistance heater (not illustrated) and a temperature sensor 41 which are built-in and outputs the sensed temperature of the hot plate 5 to the controller 34. The controller 34 controls the temperature of the hot plate 5 by using the electrical resistance heater based on the sensed temperature of the hot plate 5.  
25 Incidentally, the hot plate may be, for example, a jacket having a hollow portion, to thereby heat the wafer W by supplying a heat medium circulatingly to the hollow portion.

A mass flow controller 42, for example, is

provided to the exhaust pipe 17 and the controller 34 controls the exhaust flow.

5 A pressure sensor 43, for example, is attached inside the treatment chamber 7 and the sensed pressure inside the treatment chamber 7 by this pressure sensor 43 is outputted to the controller 34. The controller 34 controls the mass flow controllers 33 and 42 based on the sensed pressure inside the treatment chamber 7. Thus, the flow of the silylation reagent vapor supplied  
10 into the treatment chamber 7 and the exhaust gas exhausted from the treatment chamber 7 are controlled.

Note that the bubbler tank 32 is not limited to the aforementioned structure, but may perform the bubbling, for example, by forming many holes in the  
15 supply pipe 36 and supplying the gas through these holes, without using the bubbling member 35 for performing the bubbling. It is preferable to provide a check-valve to the supply pipe 36 in order to prevent the backflow of the silylation reagent 38 while the gas  
20 is not supplied.

The silylation treatment unit is applied to a coating and developing system shown in FIG. 5 to FIG. 7.

As shown in FIG. 5, the coating and developing system includes a load/unload section 62 which takes  
25 the wafer W out sequentially from a cassette CR storing the wafers W, a processing section 63 which performs processing of coating a resist solution and developing



to the wafer W taken out by the load/unload section 62,  
and an interface section 64 which transfers the wafer W  
coated with the resist solution to an exposure unit not  
illustrated. The load/unload section 62 includes a  
5 mounting table 65 which takes in/out the cassette CR  
storing, for example, 25 semiconductor wafers W.

In the load/unload section 62, as shown in FIG. 5,  
a plurality of, for example, up to 4 cassettes CR are  
mounted in a line at a positioning projection 65a on  
10 the mounting table 65, in an X direction each directing  
its access ports of the wafer toward the processing  
section 63. A first sub-arm mechanism 66 which is  
movable in the cassette alignment direction (X  
direction) and in a wafer alignment direction of the  
15 wafers W stored in the cassette CR (Z direction; a  
vertical direction) is made to give access to each of  
the cassettes CR selectively.

Further, the first sub-arm mechanism 66 is  
structured rotatively in a  $\theta$  direction and is able to  
20 transfer the wafer W to a main arm mechanism 67 which  
is provided in the processing section 63. Further, as  
described later, it can give access to an alignment  
unit (ALIM) and an extension unit (EXT) which belong to  
a multi-tiered unit section of a third processing unit  
25 group G3 of the processing section 63 side.

The transfer of the wafer W between the  
load/unload section 62 and the processing section 63 is

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performed through the third processing unit group G3. This third processing unit group G3 is structured by vertically piling up a plurality of processing units as shown in FIG. 7. More specifically, the processing unit group G3 is structured by sequentially piling a cooling unit (COL) which performs the cooling treatment to the wafer W, an adhesion unit (AD) which performs the hydrophobic treatment for increasing the adhesion property of the resist solution to the wafer W, a silylation treatment unit (SLL) which performs the silylation treatment to the wafer W, a dry developing unit (DDEV) which performs the dry developing to the silylation treated wafer W, the alignment unit (ALIM) which performs alignment of the wafer W, the extension unit (EXT) which makes the wafer W to wait, two prebaking units (PREBAKE) each of which performs the heating treatment before an exposure processing, two postbaking units (POBAKE) each of which performs the heating treatment after the exposure processing, and a post-exposure baking unit (PEBAKE), from bottom to top.

The transfer of the wafer W to the main arm mechanism 67 is performed through the extension unit (EXT) and the alignment unit (ALIM).

As shown in FIG. 5, first to fifth processing unit groups G1 to G5 including the above third processing unit group G3 are provided around the main arm mechanism 67 in such a manner to surround the main arm

mechanism 67. Similarly to the third processing unit group G3, other processing unit groups G1, G2, G4 and G5 are structured by vertically piling up the various processing units. The silylation treatment unit (SLT) of the present invention is provided to the third and fourth processing unit groups G3 and G4.

Meanwhile, as shown in FIG. 7, the main arm mechanism 67 is equipped with a main arm 68 which can ascend and descend freely in a vertical direction ( $Z$  direction) inside a cylindrical guide 69 connected extendedly in a vertical direction. The cylinder-shaped guide 69 is connected to a rotation shaft of a motor (not illustrated) and rotates integrally with the main arm 68 around the rotation shaft by the rotation power of the motor, whereby the main arm 68 is made rotatively in a  $\theta$  direction. The cylinder-shaped guide 69 may be structured in such a manner to connect to another rotation shaft (not illustrated) which rotates by the above motor. As described above, the main arm 68 is driven vertically so that the wafer W is able to arbitrarily give access to each processing unit of each of the processing unit groups G1 to G5.

The main arm mechanism 67 which receives the wafer W through the extension unit (EXT) of the third processing unit group G3 from the load/unload section 62, first carries this wafer W into the adhesion unit (AD) of the third processing unit group G3 to perform

the hydrophobic treatment. Next, the wafer W is carried out from the adhesion unit (AD) and subjected to the cooling treatment in the cooling unit (COL).

The wafer W after the cooling treatment is opposingly positioned to a resist solution coating unit (COT) of the first processing unit group G1 (or the second processing unit group G2) by the main arm mechanism 67 and carried thereinto.

The wafer W coated with the resist solution is unloaded by the main arm mechanism 67 and transferred to the interface section 64 through the fourth processing unit group G4.

As shown in FIG. 7, this fourth processing unit group G4 is structured by sequentially piling the cooling unit (COL), an extension-cooling unit (EXT-COL), the extension unit (EXT), the cooling unit (COL), the silylation treatment unit (SLL), the dry developing unit (DDEV), two prebaking units (PREBAKE), and three postbaking units (POBAKE) from bottom to top.

The wafer W carried out from the resist solution coating unit (COT) is first inserted into the prebaking unit (PREBAKE) for removing a solvent (thinner) from the resist solution and drying. This drying can be performed, for example, by means of a reduced pressure method. That is, it may be the method in which the wafer W is inserted into the prebaking unit (PREBAKE) or a chamber separately provided therefrom so that the

solvent is removed (the resist solution is dried) by reducing the pressure in the periphery around the wafer W.

5       Next, the wafer W is transferred to a second sub-arm mechanism 70 which is provided in the interface section 64 through the extension unit (EXT) after this wafer W is cooled in the cooling unit (COL).

10       The second sub-arm mechanism 70 which has received the wafer W transfers the received wafer W into a buffer cassette BUCR sequentially. The interface section 64 transfers this wafer W to the exposure unit not illustrated and receives the exposure processed wafer W.

15       The wafer W after the exposure processing is transferred to the main arm mechanism 67 through the fourth processing unit group G4 in the reverse order to the above after an unnecessary resist film in the peripheral portion of the wafer is removed by a peripheral exposure unit (WEE), in which this main arm  
20       mechanism 67 carries the exposure processed wafer W into the silylation treatment unit (SLL). The wafer W which has been subjected to the silylation treatment in the silylation treatment unit (SLL) is carried into the dry developing unit (DDEV) to be subjected to the dry  
25       developing. Subsequently, it is transferred out to the load/unload section 62 through the extension unit (EXT).

      Note that the fifth processing unit group G5 is

selectively provided and it is structured similarly to the fourth processing unit group G4 in this embodiment. Further, the fifth processing unit group G5 is movably held by a rail 71 so that the maintenance processing of the main arm mechanism 67 and the first to fourth processing unit groups G1 to G4 can be easily performed.

When the silylation treatment unit of the present invention is applied to a coating and developing unit shown in FIG. 5 to FIG. 7, it can remarkably decrease the area for installation of the unit because each processing unit is structured by piling up vertically.

It is a matter of course that the silylation treatment unit shown in this embodiment can be applied to the units other than the coating and developing unit like this. Further, various changes may be made therein without departing from the spirit of the present invention.

Next, the silylation treatment process by the coating and developing system is explained with reference to process sectional views in FIGS. 8A to 8C and a flow chart in FIG. 9.

When a main switch of the coating and developing system is turned on, electric power supply begins respectively from each power source to the silylation treatment unit 1.

Then, the shutter 13 is opened and the wafer W is mounted on an arm holder (not illustrated) for holding

the main arm of the main arm mechanism 67, which is inserted into the treatment chamber 7. When carrying in the wafer W, the lifter pins 10 are raised from the hot plate 5 by about 18 mm (S1), the wafer W is transferred from the arm holder to the lifter pins 10, and the arm holder is retreated from the treatment chamber 7 (FIG. 8A, S2). Since the temperature inside the treatment chamber 7 when carrying in the wafer W is room temperature, it is certain that a silylation reaction on the surface of the wafer W does not occur at this stage.

After the wafer W is carried into the position spaced from the hot plate 5 by about 18 mm, the shutter 13 is raised to seal up the treatment chamber 7 and the atmosphere is exhausted from the exhaust port 17 to reduce the pressure inside the treatment chamber 7 (S3). Further, when the pressure is reduced to a predetermined value, for example, 80 pascals, the silylation reagent vapor is supplied from the supply holes 14a (S4). Here, the temperature of the silylation reagent vapor is preferably made nearly the same as that of the wafer W, for example, about 40°C to about 50°C in order to avoid the reaction from progressing unexpectedly.

Then, when the treatment chamber 7 is filled with the silylation reagent vapor, the hot plate 5 is heated (S5). Further, the lifter pin 10 is descended to set

the interval between the wafer W and the hot plate 5 by about 7 mm (S6). Incidentally, the hot plate 5 may of course be heated after the lifter pin 10 is descended. The wafer W is spaced from the hot plate 5 so that the temperature of the wafer W is kept lower than that of the surface of the hot plate 5. Specifically, the temperature of the wafer W is held about 40-50°C in order to keep the temperature of the surface of the wafer W to an extent not to cause the silylation reaction to occur (FIG. 8B). Under this temperature condition, it is necessary to wait until the silylation reagent vapor is uniformly dispersed inside the treatment chamber 7 (S7). When the silylation reagent vapor is uniformly dispersed, the temperature of the wafer W becomes uniform in its surface.

Moreover, the hot plate 5 is kept heating in the conditions where the treatment chamber 7 is sealed up to maintain a constant pressure while the carrying in of the silylation reagent vapor is stopped and the exhaust of the gas from the exhaust pipe 17 is stopped so that all the gas flow inside the treatment chamber 7 is stopped, and where the temperature inside the treatment chamber 7 is made uniform in the surface of the wafer W (S8).

Furthermore, when the temperature of the hot plate 5 itself becomes, for example, about 80-90°C, the lifter pins 10 are further descended so that the



interval between the wafer W and the hot plate 5 is set to be about 0.1 mm (FIG. 8C, S9). Here, as the wafer W is heated to about 80-90°C, the temperature of the wafer W also rises to 80-90°C and the silylation progresses on the surface thereof (S10). The silylation progresses uniformly in the surface of the wafer W because the carrying in of the gas into the treatment chamber 7 and the exhaust of the gas from the treatment chamber 7 are already stopped to obtain sealing, and the planer uniformity of the temperature inside the treatment chamber 7 is maintained in this silylation.

When the silylation is completed, the N<sub>2</sub> gas is supplied into the treatment chamber 7 from the gas supply port which is not illustrated while the gas including the silylation reagent vapor is exhausted from the exhaust port 17 so that the gas inside the treatment chamber 7 which is the silylation reagent vapor is replaced by the N<sub>2</sub> gas in order to for the silylation reaction to finish (S11). As the silylation reaction takes several seconds, the silylation reaction finishes immediately by replacing the gas inside the treatment chamber 7. Incidentally, until the temperature of the surface of the wafer W lowers to about 50°C while replacing the gas, it is necessary to maintain the uniform temperature on the surface of the wafer W because the silylation reaction progresses

although slowly. Thus, it is preferable to have a larger interval between the wafer W and the hot plate 5 than 7 mm by ascending the lifter pins 10 before replacing the gas. Further, it is preferable that the N<sub>2</sub> gas to be supplied has a lower temperature than about 50°C which is the critical temperature at which the silylation reaction occurs. The gas may of course be replaced without raising the lifter pins 10, in which the interval from the hot plate 5 is 0 mm.

According to the aforementioned process, the following effects can be obtained.

First, by making the wafer W to wait with a predetermined interval from the hot plate 5 from the silylation reagent vapor is carried into the treatment chamber 7 until the silylation reagent vapor disperses uniformly therein, it is possible to keep the wafer W at a temperature of 50°C or less during waiting, at which the silylation reaction does not occur so that the vapor density inside the treatment chamber 7 can become uniform during this waiting time. The silylation reaction has a temperature dependency, in which the high silylation rate is obtained at the higher temperature. The silylation treatment can be performed with the high planar uniformity of the wafer W by maintaining the uniform temperature in the surface of the wafer W like this.

Second, by setting the interval between the hot

plate 5 and the wafer W to the three levels, the stable  
silylation treatment can be performed without being  
affected by the transitional temperature change until  
the temperature of the wafer W rises high enough where  
5 the silylation reaction progresses. That is, although  
the temperature variability of the wafer W is very  
large while the temperature of the wafer W rises from  
23°C to 80°C, the temperature variability becomes  
comparatively smaller once it has risen to 50°C and  
10 further rises to about 80°C to 90°C so that the  
silylation treatment with smaller temperature  
variability becomes possible. Further, the silylation  
treatment with enough stability can be simply performed  
only by setting the interval between the hot plate 5  
15 and the wafer W to the plural levels to perform the  
treatment, even if there are some problems in the  
structure of the hardware. For example, when the  
silylation reagent is supplied from the upper portion  
of the chamber by using a shower or the like, the shape  
20 of the blow-out ports of the silylation reagent needs  
to be extremely precise, but the stable silylation  
treatment can be performed according to the present  
invention, regardless of the structure of the blow-out  
ports.

25 Third, since the silylation treatment is performed  
under the sealed up space inside the treatment chamber  
7 without reducing the pressure and exhausting, the

variability in the density can be limited to a minimum.

Fourth, the wafer W may be lifted up after the silylation reagent vapor is completely exhausted. It is possible to prevent the reaction from progressing  
5 unexpectedly on the surface of the wafer W by the method like this.

Fifth, when replacing the silylation reagent vapor by the N<sub>2</sub> gas after the completion of the silylation treatment, which is performed by once spacing the wafer  
10 W from the hot plate 5 at a predetermined interval, so that the ununiform silylation reaction can be prevented because the gas can be replaced after setting the wafer W at the temperature at which the silylation reaction is difficult to progress.

Sixth, when the silylation reagent vapor is supplied to obtain uniformity, which is performed after its pressure is once reduced, the amount of gas  
15 existing in the treatment chamber 7 decreases so that the uniformity of the density of the silylation reagent improves.  
20

Incidentally, the silylation treatment unit according to the present invention can be applied to the other structures than that of this embodiment.

First, a silylation reagent vapor supply system is  
25 not limited to the structure shown in FIG. 2. The supply system can take any structures, for example, the structure in which one or plural holes is/are provided

in the hot plate 5, through which the silylation reagent vapor is supplied into the surface side of the hot plate 5, and the structure in which a supply port is provided near the exhaust port 17, through which it is supplied toward the treatment chamber 7.

Second, the lifter pin 10 which holds the wafer W at a predetermined interval to the hot plate 5 can move between the plural levels, besides the three levels of the high level, the medium level and the low level. Further, it can move continuously from the high level to the medium level, or from the medium level to the low level.

FIG. 10 shows an embodiment where the hot plate 5 is held at more than four levels. FIG. 10 shows the case where the wafer W moves between the five levels. The treatment in the high level where the wafer W is carried in (the interval between the hot plate 5 and the wafer W is, for example, 18 mm) and the treatment in the low level where the treatment chamber 7 is sealed to promote the silylation treatment (the interval between the hot plate 5 and the wafer W is 0 mm) are the same as the case of the three levels (S1 to S7, S8 to S11). In this embodiment, the hot plate 5 is gradually moved downward in the plural levels when making the temperature variability inside the treatment chamber 7 uniform.

In this case, the wafer W is once held at the

position lower than the high level (the interval between the hot plate 5 and the wafer W is, for example, 7 mm, S6) until the temperature of the wafer W rises to a predetermined temperature (50°C, for example). The  
5 wafer W is moved downward (the interval between the hot plate 5 and the wafer W is, for example, 5 mm, S21 and S22) when the temperature inside the treatment chamber 7 rises (60°C, for example). The wafer W is further moved downward (the interval between the hot plate 5  
10 and the wafer W is, for example, 3 mm, S23 and S24) after the temperature further rises (70°C, for example).

The silylation reagent vapor is continuously supplied thereinto similarly to the case shown in FIG. 9 until the interval between the hot plate 5 and  
15 the wafer W becomes 7 mm to 0 mm. Thus, the wafer W is moved downward in the plural levels to the low level finally, in which the wafer W contacts the hot plate 5. Accordingly, the wafer W can move corresponding to the change of the temperature variability, compared with  
20 the case where it is held only at one level of the medium level. Therefore, the silylation treatment with the reduced influence of the temperature variability can be performed.

Note that the present invention can of course be  
25 applied to the case with four levels or six or more levels although the example of the wafer W which moves vertically in five levels is illustrated in FIG. 10.

Further, the interval between the hot plate 5 and the wafer W or the temperature inside the treatment chamber 7 when the wafer W is gradually moving is an example, which is not limited to the above.

5 Further, the structure of the supply ring 14 can be modified to use as follows. In the supply ring 14 shown in FIG. 11, supply holes 14p, 14q and 14r are formed in the inner circumferential surface of the ring member 14b, in which the diameters of the supply holes  
10 are set to become larger as their positions become higher. That is, the diameter of the supply hole 14p which is positioned at the lowermost is the smallest, the diameter of the supply hole 14q which is positioned above is larger than that of the supply hole 14p, and  
15 the diameter of the supply hole 14r which is positioned at the uppermost is set to be the largest. Thus, by setting the diameters of the supply holes which are vertically arrayed in the inner circumferential surface of the ring member 14b to become larger as the holes  
20 positioned higher, the increased volume of the vapor can be supplied to the surface of the wafer W to be subjected to the silylation treatment so as to perform the treatment more effectively.

25 Furthermore, in the supply ring 14 shown in FIG. 12, a plurality of the supply holes 14a are formed in almost half of the inner circumferential surface of a ring member 14b, and a plurality of exhaust holes 14e

are formed opposingly to the supply holes 14a in the remaining half of the inner circumferential surface. Thus, when the supply hole 14a and the exhaust hole 14e are opposingly disposed like this, the vapor supplied from the supply hole 14a is exhausted as it is, from the opposing exhaust hole 14e by flowing horizontally. Therefore, turbulence does not occur so that the uniformity of the treatment is improved and an excellent exhaust efficiency is obtained.

The holes 9 for vertically moving the lifter pins 10 are formed in the hot plate 5. As shown in FIG. 14, the inert gas, for example, the  $N_2$  gas may be blew out through the holes 9 to the non-treatment surface of the wafer W, for example, the back surface of the wafer W. In the example shown in FIG. 14, a gas supplying section 81 and a pipeline 82 are provided for supplying the  $N_2$  gas to the holes 9.

It is preferable to start blowing out the  $N_2$  gas to the back surface of the wafer W like this when the silylation treatment is completed and the vapor including the silylation reagent inside the chamber 7 is purged by the inert gas, or when the vapor including the silylation reagent is purged from inside the chamber 7 by the inert gas and the wafer W starts being lifted by the lifter pins 10 as shown in FIG. 15.

It is possible to prevent a deposition from adhering to the non-treatment surface of the wafer W by



blowing out the gas to the non-treatment surface of the wafer W, for example, to the back surface of the wafer W. The supply ring 14 shown in FIG. 16 is structured so that upper supply holes, for example, 14l and 14m supply a processing gas, for example, the vapor including the silylation reagent to the treatment surface of the wafer W, while the lower supply hole 14n blows out the inert gas, for example, the N<sub>2</sub> gas to the non-treatment surface of the wafer W, among the supply holes 14l, 14m and 14n which are arrayed vertically in the inner circumferential surface of the ring member 14b. Thus, it is possible to supply the processing gas, for example, the vapor including the silylation reagent to the treatment surface of the wafer W, and blow out the inert gas, for example, the N<sub>2</sub> gas to the non-treatment surface of the wafer W. Therefore the adhesion of the deposition to the non-treatment surface of the wafer W can be prevented.

Incidentally, the upper supply holes 14l and 14m may have the structures in which the processing gas, for example, the vapor including the silylation reagent and the inert gas can be selectively blown out.

Additional advantages and modifications will readily occur to those skilled in the art. Therefore, the invention in its broader aspects is not limited to the specific details and representative embodiments shown and described herein. Accordingly, various

modifications may be made without departing from the spirit or scope of the general inventive concept as defined by the appended claims and their equivalents.

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WHAT IS CLAIMED IS:

1. A unit for performing a silylation treatment  
on the surface of a substrate, comprising:

a chamber;

5 a heating mechanism provided in said chamber for  
heating the substrate;

a supplying mechanism for supplying a vapor  
including a silylation reagent into said chamber; and

10 a substrate holder for holding the substrate in  
said chamber, in which an interval between said heating  
mechanism and the substrate is adjustable to at least  
three levels or more.

15 2. The unit according to claim 1, wherein the  
temperature of the vapor is set to be almost the same  
as that of the substrate.

3. The unit according to claim 1, wherein the  
vapor is supplied horizontally.

20 4. The unit according to claim 1, wherein said  
supplying mechanism is a supply ring in a ring shape  
for surrounding the substrate.

5. The unit according to claim 4, wherein a  
plurality of supply holes are formed in an inner  
circumferential surface of a ring member of the supply  
ring.

25 6. The unit according to claim 5, wherein the  
supply holes are also disposed in a vertical direction  
of the inner circumferential surface, in which a

diameter of the upper supply hole is larger than a diameter of the lower supply hole.

7. The unit according to claim 4, wherein a plurality of supply holes are formed in almost half of an inner circumferential surface of a ring member of the supply ring and a plurality of exhaust holes are formed opposingly to the supply holes in the remaining half of the inner circumferential surface.

8. The unit according to claim 1, wherein said supplying mechanism supplies the vapor only to a treatment surface of the substrate.

9. The unit according to claim 8, wherein said supplying mechanism supplies an inert gas to a non-treatment surface of the substrate.

10. The unit according to claim 9, wherein said supplying mechanism is structured to be able to supply the inert gas to the treatment surface of the substrate selectively.

11. The unit according to claim 8, further comprising:

a hot plate as said heating mechanism;

lifter pins as said substrate holder; and

holes formed penetratingly through the hot plate in a vertical direction, through which the lifter pins project from the hot plate for moving in a vertical direction,

wherein an inert gas is supplied to a

non-treatment surface of the substrate through the holes.

12. A method for performing a silylation treatment on the surface of a substrate, comprising the steps of:

5 carrying in the substrate into a chamber and disposing it at a predetermined interval from a heating mechanism provided in said chamber;

supplying a vapor including a silylation reagent into said chamber such that said chamber is filled with  
10 an atmosphere of the silylation reagent;

raising the temperature in said chamber by said heating mechanism;

bringing the substrate closer to said heating mechanism such that the silylation atmosphere is  
15 dispersed uniformly inside said chamber at a temperature where a silylation reaction of the substrate does not occur; and

further bringing the substrate closer to said heating mechanism to make the temperature of the  
20 substrate higher such that the silylation reaction occurs on the surface of the substrate.

13. The method according to claim 12, wherein an interval between said heating mechanism and the substrate is adjustable to at least three levels or  
25 more.

14. The method according to claim 12, wherein the vapor including the silylation reagent is supplied

after the pressure of said chamber is reduced.

15. The method according to claim 12, wherein the silylation reaction is made to occur where the supply of the vapor including the silylation reagent into said chamber is stopped, the exhaust from said chamber is not performed, and said chamber is sealed.

16. The method according to claim 12, wherein the silylation reaction is stopped by supplying an inert gas into said chamber and exhausting the vapor including the silylation reagent from said chamber.

17. The method according to claim 16, wherein the substrate is carried away from said heating mechanism after the vapor including the silylation reagent is exhausted from said chamber by supplying the inert gas.

ABSTRACT OF THE DISCLOSURE

A silylation treatment unit includes a chamber, a heating mechanism provided in this chamber for heating a substrate, a supplying mechanism for supplying a vapor including a silylation reagent into the chamber. The unit also has a substrate holder for holding the substrate in the chamber, in which an interval between the heating mechanism and the substrate is adjustable to at least three levels or more. The substrate is received such that it is least influenced by a heat in the chamber by maximizing the interval from the heating mechanism. The interval is brought comparatively closer to the heating mechanism to wait until the temperature inside the chamber obtains a high planer uniformity. The interval is brought further closer to the heating mechanism after a high planer uniformity is obtained such that a silylation reaction occurs.

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FIG.1

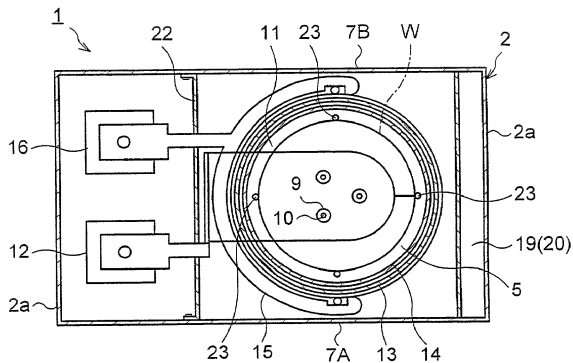
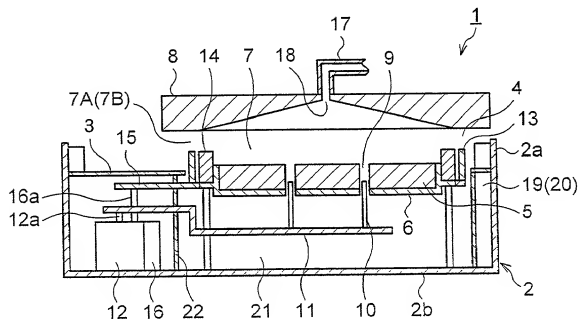


FIG.2





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FIG.3

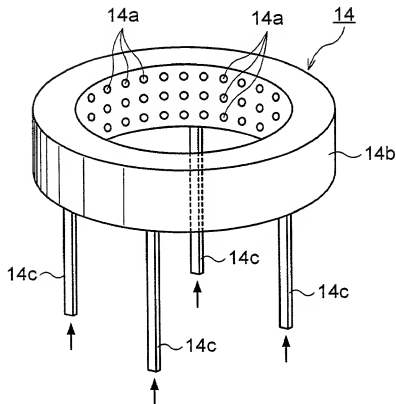
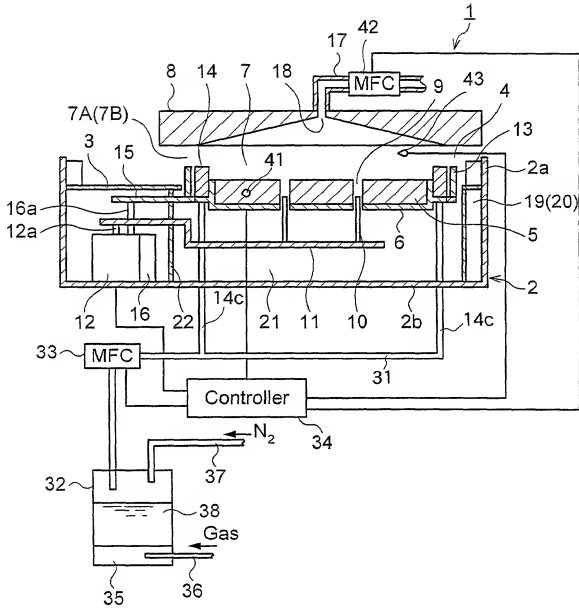
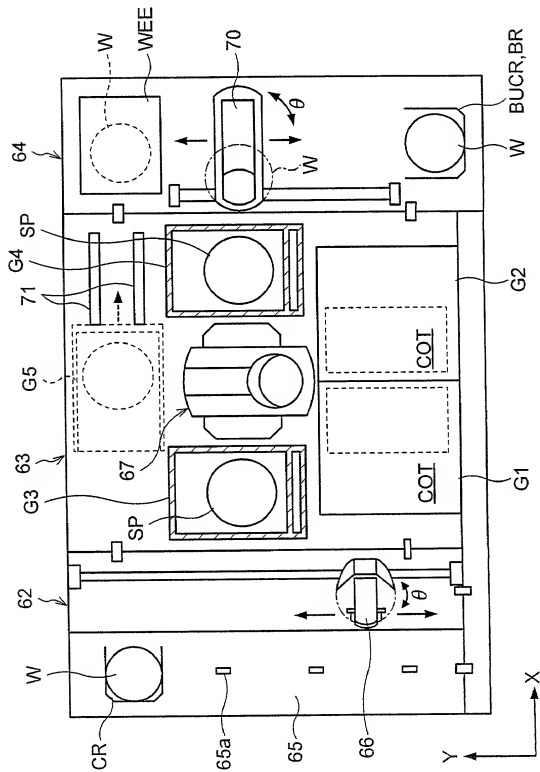


FIG.4

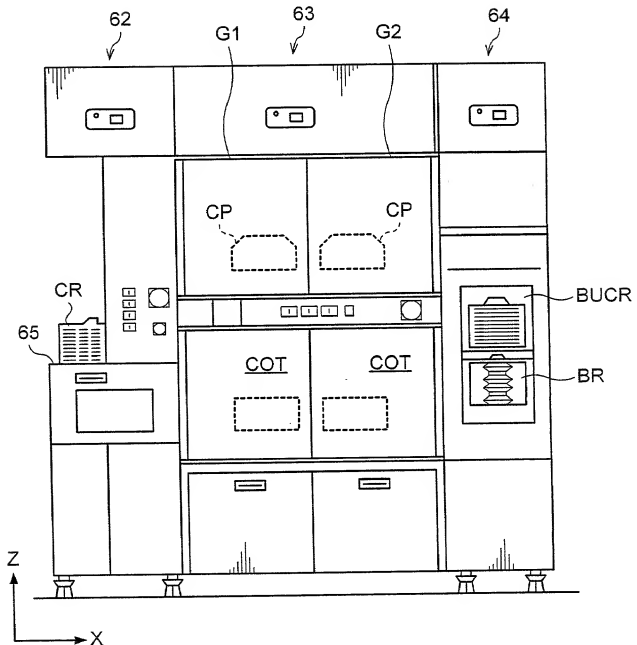


**FIG. 5**



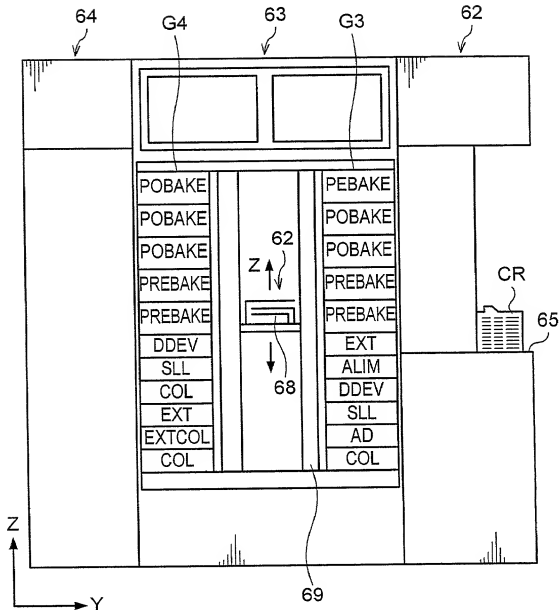
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FIG.6



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FIG. 7



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FIG.8A

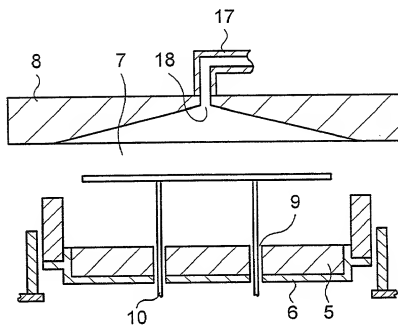
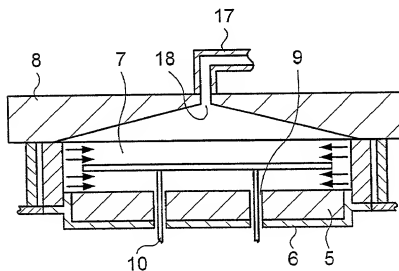
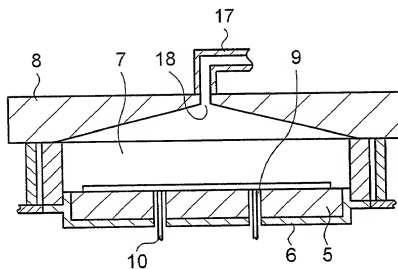


FIG.8B



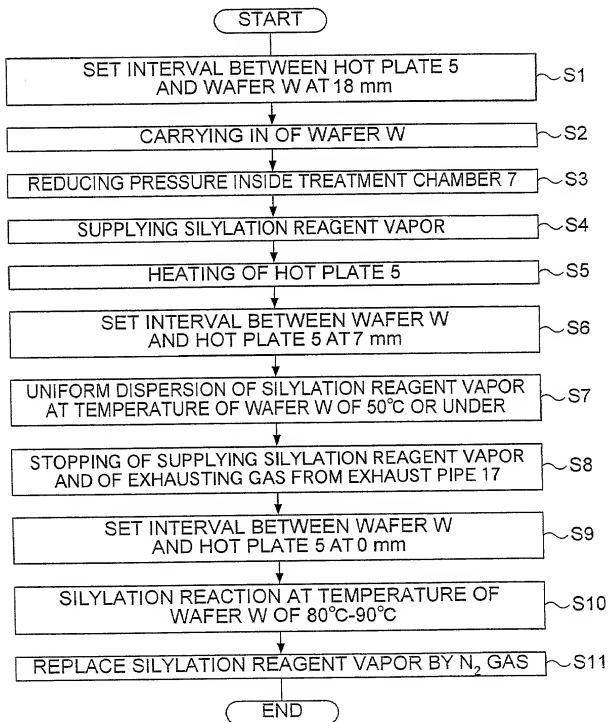
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FIG.8C



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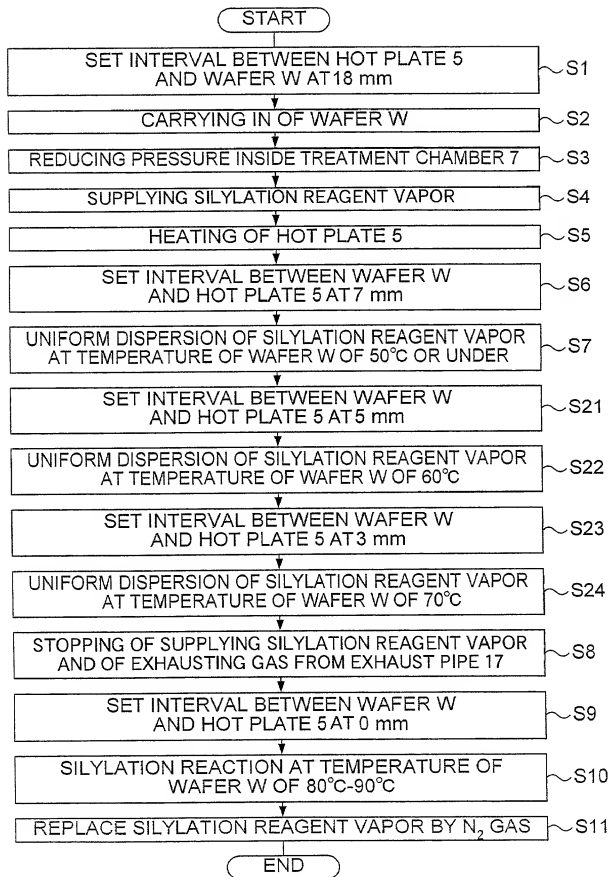
FIG.9





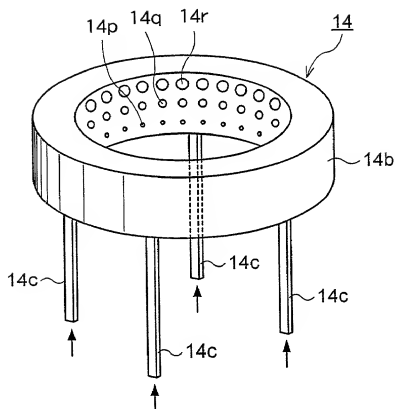
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FIG.10



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FIG.11



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FIG.12

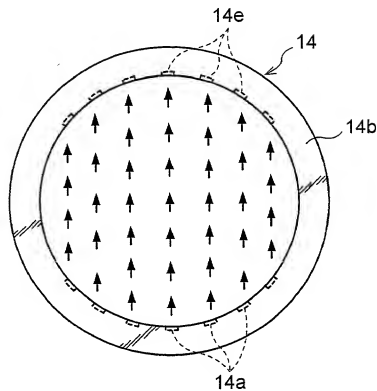
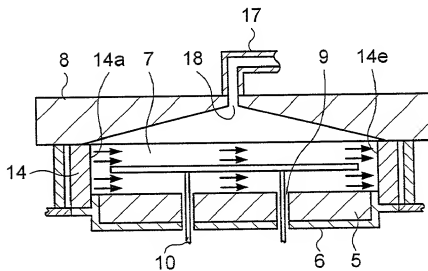


FIG.13



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FIG.14

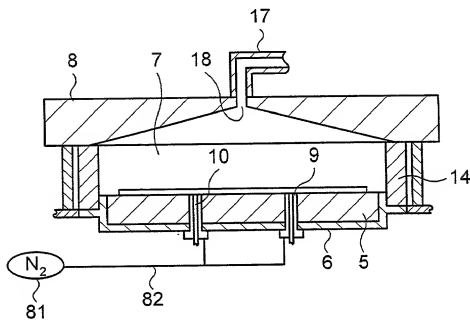
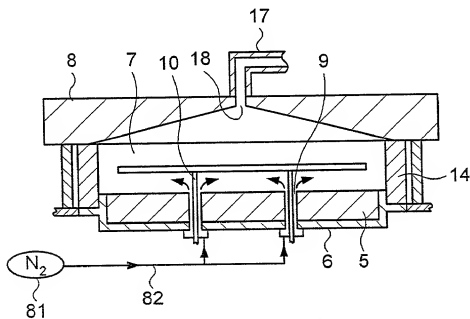
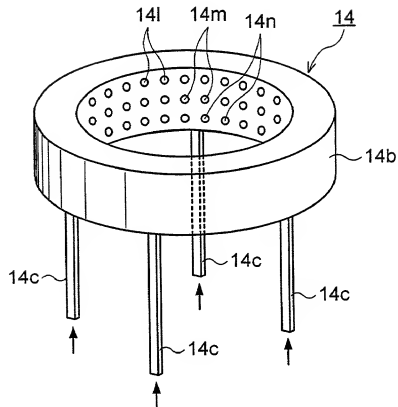


FIG.15



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FIG.16



# Declaration Power of Attorney For Patent Application

## 特許出願宣言

### Japanese Language Declaration

私は、下欄に氏名を記載した発明として、以下の通り宣言する：

私の住所、郵便の宛先および国籍は、下欄に氏名に続いて記載したとおりであり、

名称の発明に関し、請求の範囲に記載した特許を求める主題の本来の、最初にして唯一の発明者である（一人の氏名のみが下欄に記載されている場合）か、もしくは本来の、最初にして共同の発明者である（複数の氏名が下欄に記載されている場合）と信じ、

シリル化処理装置及び方法

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その明細書を  
（該当するほうに印を付す）

☐ ここに添付する。

☐ \_\_\_\_\_月 \_\_\_\_\_日に

出願番号第 \_\_\_\_\_ 号として

提出し、 \_\_\_\_\_ 月 \_\_\_\_\_ 日に補正した。  
（該当する場合）

私は、前記のとおり補正した請求の範囲を含む前記明細書の内容を検討し、理解したことを陳述する。

私は、連邦規則法典第37部第1章第56条（a）項に従い、本願の審査に所要の情報を開示すべき義務を有することを認める。

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

SILYLATION TREATMENT UNIT AND METHOD

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The specification of which  
(check one)

☒ is attached hereto.

☐ was filed on \_\_\_\_\_  
as Application Serial No.

\_\_\_\_\_ and was amended on

\_\_\_\_\_ (if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, Section 1.56(a).

# Japanese Language Declaration

私は、合衆国法典第35部第119条、第172条、又は第365条に基づく下記の外国特許出願又は発明者証出願の外国優先権利益を主張し、さらに優先権の主張に係る基礎出願の出願日等の出願日を有する外国特許出願又は発明者証出願を以下に明記する：

I hereby claim foreign priority benefits under Title 35, United States Code Sec. 119, Sec. 172 or Sec. 365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior foreign application(s)  
先の外国出願

Priority Claimed  
優先権の主張

11-328269  
(Number)  
(番号)

JAPAN  
(Country)  
(国名)

18/11/1999  
(Day/Month/Year Filed)  
(出願年月日)

☒ ☐  
Yes No  
あり なし  
☐ ☐  
☐ ☐  
☐ ☐  
☐ ☐

私は、合衆国法典第35部第120条に基づく下記の合衆国特許出願の利益を主張し、本願の請求の範囲各項に記載の主題が合衆国法典第35部第112条第1項に規定の態様で先の合衆国出願に開示されていない限りにおいて、先の出願の出願日と本願の国内出願日又はPCT国際出願日の間に公表された連邦規則法典第37部第1章第56条(a)項に記載の所要の情報を開示すべき義務を有することを認める。

I hereby claim the benefit of Title 35, United States Code, Sec. 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Sec. 112, I acknowledge the duty to disclose any material information as defined in Title 37, Code of Federal Regulations, Sec. 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application No.)  
(出願番号)

(Filing Date)  
(出願日)

(Status: Patented, Pending, Abandoned)  
(現況：特許許可済、係属中、放棄済)

(Application No.)  
(出願番号)

(Filing Date)  
(出願日)

(Status: Patented, Pending, Abandoned)  
(現況：特許許可済、係属中、放棄済)

私は、ここに自己の知識に基づいて行った陳述がすべて真実であり、自己の所有する情報及び信ずるところに従って行った陳述で真実であると信じ、更に故意に虚偽の陳述等を行った場合、合衆国法典第18部第1001条により、罰金もしくは禁錮に処せられるか、又はこれらの刑が併科され、又はかかる故意による虚偽の陳述が本願ないし本願に対して付与される特許の有効性を損なうことがあることを認識して、以上の陳述を行ったことを宣言する。

I hereby declare that all statements made herein of my own knowledge are true; and further that all statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

0973247-11500

# Japanese Language Declaration

(日本語宣言書)

委任状: 私は、下記の発明者として、本出願に関する一切の手續きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。  
(弁理士、または代理人の氏名及び登録番号を明記のこと)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (list name and registration number)

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